

# FDTs as dewetting coating for an Electrowetting controlled silicon photonic switch

Sarah Günther<sup>1</sup>, Shuhao Si<sup>1</sup>, Herbert D'heer<sup>2</sup>, Dries van Thuuthout<sup>3</sup> and Martin Hoffmann<sup>4</sup>

<sup>1</sup>Micromechanical Systems Group, Technische Universität Ilmenau, Germany

<sup>2</sup>TOMRA Sorting Solutions, Leuven, Belgium

<sup>3</sup>Photonics Research Group, Ghent University – IMEC, Belgium

<sup>4</sup>Chair of Microsystems Technology, Ruhr-Universität Bochum, Germany

## Abstract

The self-assembled monolayer FDTs (1H,1H,2H,2H-Perfluorodecyltrichlorosilane) is presented as suitable dewetting coating material for an electrowetting on dielectrics (EWOD) controlled silicon photonics switch deployed in fiber optic telecommunication systems. The anti-sticking characteristics of FDTs are compared to those of Polytetrafluoroethylene (PTFE) as most common dewetting coating for EWOD devices. It is shown that FDTs could outperform other materials such as PTFE when an extremely thin, long-term stable and uniform layer is required. In the specific case, FDTs is applied in vapor phase as anti-sticking coating to the active optical surface of the integrated silicon photonics switch thus enabling the EWOD driven liquid motion. The suitability of the coating is presented by contact angle measurements and durability tests carried out with the switching liquids. Finally, it is demonstrated by optical measurements that the FDTs coating has a neglectable influence on the optical switching performance.

## Keywords

Couplers, integrated optics, liquids, optical fiber communication, optical switches, silicon photonics, EWOD, Electrowetting, Optofluidics

## 1. Introduction

The material Perfluorodecyltrichlorosilane (FDTs) has been widely applied for anti-sticking coating in Nanoimprint Lithography (NIL) [1][2][3][4][5]. The FDTs coating is acknowledged by its processing simplicity that requires uncomplicated facilities at wide-range temperature conditions. The monolayer coating in a thickness of single molecule suits applications in terms of optical compatibility. Its long-tail consisting of 17 fluorine atoms provides convenient anti-sticking properties. Therefore, these significant properties of FDTs make it advantageous in additional fields as well, such as electrowetting on dielectrics (EWOD) [6] or microfluidics in general [1][7][8]. In this paper, the application of the self-assembly silane as dewetting coating in an EWOD controlled silicon photonics switch is demonstrated.

A schematic illustration of the working principle of the EWOD driven silicon photonics switch is given in Fig. 1. The switch consists of an adiabatic coupler where the oxide cladding above one of the waveguides is removed. Switching is realized by exposing this waveguide to liquids with contrastive refractive indices. The respective liquid serves as a cladding material and hence by exchanging the liquids the effective index of the exposed waveguide changes [9][10]. The liquid motion over the coupler is driven by EWOD. All details on the EWOD system design, material selection and the liquid switching process are reported in [11]. To enable the switching, two fluids with a specific contrast in refractive index are exchanged over the coupler. By moving the fluids, the coupler switches between two states, the cross state and bar state. The active EWOD component that responds to the electric field and is moved is a polar fluid. The ambient liquid whereas is non-polar and does not respond to electrowetting but is displaced due to the motion of the polar liquid. In Fig. 2, a sequence of images illustrates the switching process. In Fig. 2a, the droplet is parked in position 1, a position which covers the optical sensitive switching area. Fig. 2b shows the droplet transition from position 1 to position 2 by sequential activations of the EWOD electrodes. The optical switching area is covered by the ambient liquid and the switching process is completed as shown in Fig. 2c. Due to fluidic barriers, each droplet position is physically stable which leads to a non-volatile optical switch.

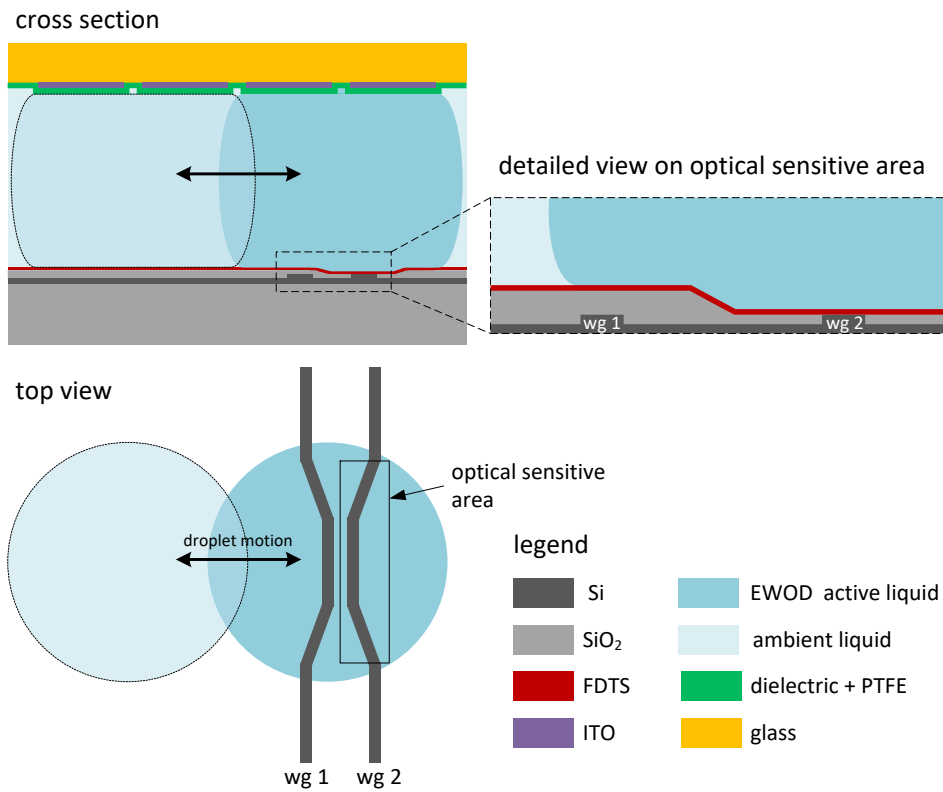


Fig. 1: Schematic diagram illustrating the working principle of the optical switch

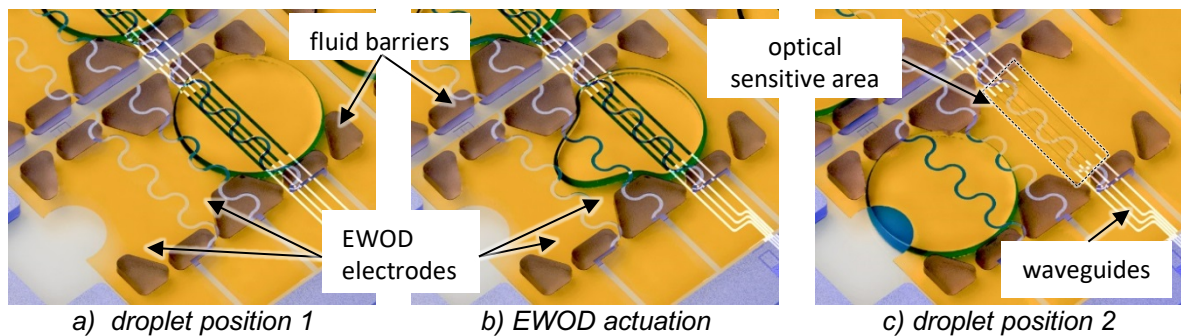


Fig. 2: Rendered 3D images of droplet motion during EWOD actuation

## 2. Material and methods

### a) Dewetting coating

Depending on the surface of the optical switch and the used liquid combination, a certain Young contact angle  $\theta_Y$  forms at the interface between the liquid and the surface of the coupler. The contact angle can be used as an indicator for the wetting behavior of the liquid. A contact angle below  $90^\circ$  corresponds to a high wettability [12], i.e. the liquid wets the surface and movement is impeded. In order to enable droplet movement, it is necessary to achieve a low wettability (contact angle higher than  $90^\circ$ ) for the active polar liquid on top of the coupler which can only be achieved by applying a dewetting coating on the optical surface. The coating material has to fulfill the anti-sticking requirement for the EWOD system as well as its optical specifications. The intensity of the optical mode of the light propagating through a waveguide decreases exponentially. Thus, the thickness of the dewetting layer is mandatorily minimized, as it greatly impacts the efficiency in the optical sensitive area. The coating should also possess a long-term stability and temperature sustainability to endure the droplet manipulation at ambient conditions.

The most commonly acknowledged dewetting layer used in electrowetting devices is Polytetrafluoroethylene (PTFE, commonly referred to the trade name Teflon™ AF). That material has excellent dewetting properties and can be deposited through several approaches, e.g. dip-coating, spin-coating, chemical vapor deposition or even sputtering [13][14][15][16][17][18]. However, certain characteristics of PTFE make it unsuitable for the application of the optical switch: The minimum coating thickness of a closed PTFE film is relatively large. In general, layers of a thickness from 100 nm to 1000 nm are used for microfluidic applications. In our experiments, it was observed that the PTFE dip coating process can be pushed to a minimum film thickness of 12 nm. At this condition, the PTFE coating still affects the optical characteristics of the optical switch extensively, which will be shown later. Further thinning by plasma etching led to a massive loss of the dewetting characteristics. Additionally, PTFE forms only physical bonds to the surface where it is applied to, rather than covalent bonding. The PTFE polymer is very stable and has a low surface energy resulting in a bad adhesion to the surface that it is applied to. Furthermore, the most common deposition methods are dip- or spin coating delivering satisfying results for plane substrates but insufficient uniformity and edge-coverage for surfaces with 3D-structures like fluid channels or etch grooves. Thus, the topology and shape of substrates that can be effectively coated are limited. By spin- or dip coating, uniform wetting of high aspect ratio structures such like micro-fluidic channels as well as sub-micro structures of high resolution and density is barely possible when material is coated in liquid phase [2]. Alternative deposition technologies, which deliver highly uniform coating for structured samples, like chemical vapor deposition or sputtering are feasible yet require dedicated equipment.

FDTs have shown several advantages over Teflon as anti-sticking coating material for the optical coupling surface. It can be deposited in vapor phase which leads to an optimal uniformity and an excellent edge coverage. The silane, i.e. the reactive head, bonds covalently to silicon and silicon dioxide which are the key materials for optical devices. This leads to a high chemical and mechanical stability. The long tail of the molecule including 17 fluorine atoms ensures the anti-sticking of the surface. The FDTs deposition results in a self-assembled monolayer (SAM) with a thickness of the single molecule having a length of  $\sim 1.4$  nm [1]. Fig. 3 gives a schematic illustration of the FDTs molecule and the SAM formation process. Experimentally, the FDTs is deposited in vapor phase at  $165^\circ\text{C}$  for at least 2 hours. Another major advantage of the vapor phase deposition is the feasibility to apply the coating after the switching device is assembled. The switch consists of an optical and an EWOD substrate, whereby the EWOD substrate contains openings for liquid filling with a diameter of approximately  $350\text{ }\mu\text{m}$ . The mounting of both substrates is carried out using UV-curing glue. However, anti-sticking coatings prevent the bonding of adhesive materials such as glue or

resin to the substrate hence hampering the assembly. This issue can be circumvented by coating after mounting. In case of the optical switch, vapor deposition of an assembled sample through the liquid filling openings is possible and was carried out successfully.

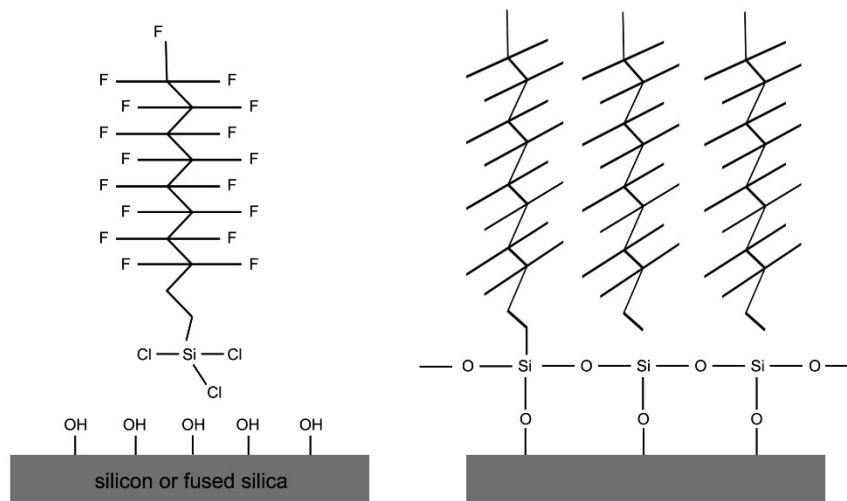


Fig. 3: Illustration of FDTD molecule and schematic sketch of the self-assembly monolayer formation process.

## b) Switching liquids

A suitable liquid combination fulfilling the complex requirements regarding both of the optical and the EWOD domains for optical coupler has been presented [11]. From the optical point of view, the liquids need to have a high contrast in refractive indices of at least 0.16 and low absorption in the telecommunication wavelength range from 1260 nm to 1650 nm. For the EWOD actuation, it is necessary to have a high polarity and high permittivity for the active liquid, while the ambient liquid should be nonpolar and have a low permittivity. The dynamic viscosities and the difference in density of the liquids are critical for the EWOD dynamics. The liquids have to retain their properties within the temperature range for ambient conditions as well. These optical, electrical and environmental demands require a novel selection of liquids listed in table 1 that has never been employed in optical or EWOD systems before. For the dewetting coating, the pair of the liquids shall meet the requirement that the contact angle for the active polar liquid should be higher than  $90^\circ$  whereas the contact angle should be below  $90^\circ$  for the ambient one. Mechanical and chemical long-term stability of the coating under exposure of the liquids should also be guaranteed.

Table 1: liquid selection for the fiber optic switch

nonpolar ambient liquid	diphenyl sulfide (DPS, CAS No. 139-66-2)
	triphenyl sulfide (TPS, CAS No. 2974-10-9)
polar electrowetting active liquid	ethylene glycol (EG, CAS No. 107-21-1)
	hydroxy propylene carbonate (HPC, CAS No. 931-40-8)

### 3. Results and discussion

The FDTD monolayer reduces surface energy due to its long fluorinated tail. The Young's equation correlates the surface energy and the contact angle  $\theta_Y$  of liquid-gas interface:

$$\gamma_{SG} = \gamma_{SL} + \gamma_{LG} \cos \theta_Y$$

where  $S$ ,  $L$ , and  $G$  refer to the solid, liquid and gas phases, respectively, and  $\gamma$  refers to the interfacial tensions of corresponding interfaces. Young's equation indicates that the reduction of surface energy can be determined by a contact angle measurement.

Contact angle measurements of each liquid on top of a silicon surface in air were performed using a Krüss DSA10 drop shape analysis system following the Young-Laplace equation [12]. The contact angles of each liquid on a PTFE coated substrate were also measured as a reference. The results of contact angle measurements can be found in Table 2. The results show that the FDTD coating exhibits an equivalent dewetting performance as PTFE.

Table 2: Measurement of contact angle following the Young-Laplace Method

Liquid		DPS	TPS	EG	HPC	H <sub>2</sub> O
CA	Air/FDTS	85	as DPS	97	99	116
	Air/PTFE	84	as DPS	99	103	116

The stability and insolubility of FDTD coating in contact with the liquids have been approved by experiment over a period of 60 days. An optical switch has been assembled and filled with the liquids HPC and TPS. EWOD actuation was tested initially and after 30 and 60 days, respectively. Droplet motion could be observed over the full test period, indicating that the FDTD layer on the silicon dioxide surface of the optical chip is stable and has not degraded. Therefore, the FDTD is approved for its stability and insolubility in the selected liquids for the application of the active optical switch.

Finally, the optical performance of an optical switch device with FDTD coating has been characterized. The transmission of light propagating through an optical switch in its cross state is measured in an optical substrate with 1) no coating applied, 2) PTFE coating in a thickness of approximately 12 nm and 3) a monolayer coating of FDTD. In each configuration, the transmission was measured for the four possible paths that light can propagate in a 2x2 optical switch, shown schematically in Fig. 4. The transmission in cross state corresponds to the paths L0-R1 and L1-R0 and in bar state to L0-R0 and L1-R1. Fig. 5 shows the measurements of the optical switch in cross state for no coating, FDTD coating and a ~12 nm PTFE coating. All of them should allow high transmission of light in the paths L0-R1 and L1-R0. This is verified for the measurements with no coating (Fig. 5a and 5c) and also for the measurements with FDTD (Fig. 5b). In the bar ports, light of the L0-R0 path (red) and L1-R1 path (blue) was significantly attenuated, whereas in the cross ports light of the L0-R1 path (yellow) and L1-R0 path (green) experienced high transmission. For the case of PTFE coating (Fig 5d), the measurements show inverted results. The cross state is prevented and light travels in the bar ports instead, i.e. the paths L0-R0 and L1-R1 show high transmission of light, while the cross ports L0-R1 and L1-R0 show attenuation. The results corresponding to the PTFE-coated substrate indicate that the use of PTFE as an anti-wetting coating in such active optical switches substantially degrades the optical performance of the switch in its cross state. The optical switch with a monolayer coating of an FDTD-treated silicon substrate, however, demonstrated that the transmission profiles in the cross state are similar to switches with no coating.

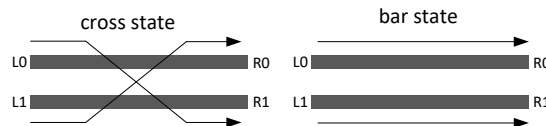


Fig. 4: Possible light paths in 2x2 optical switch



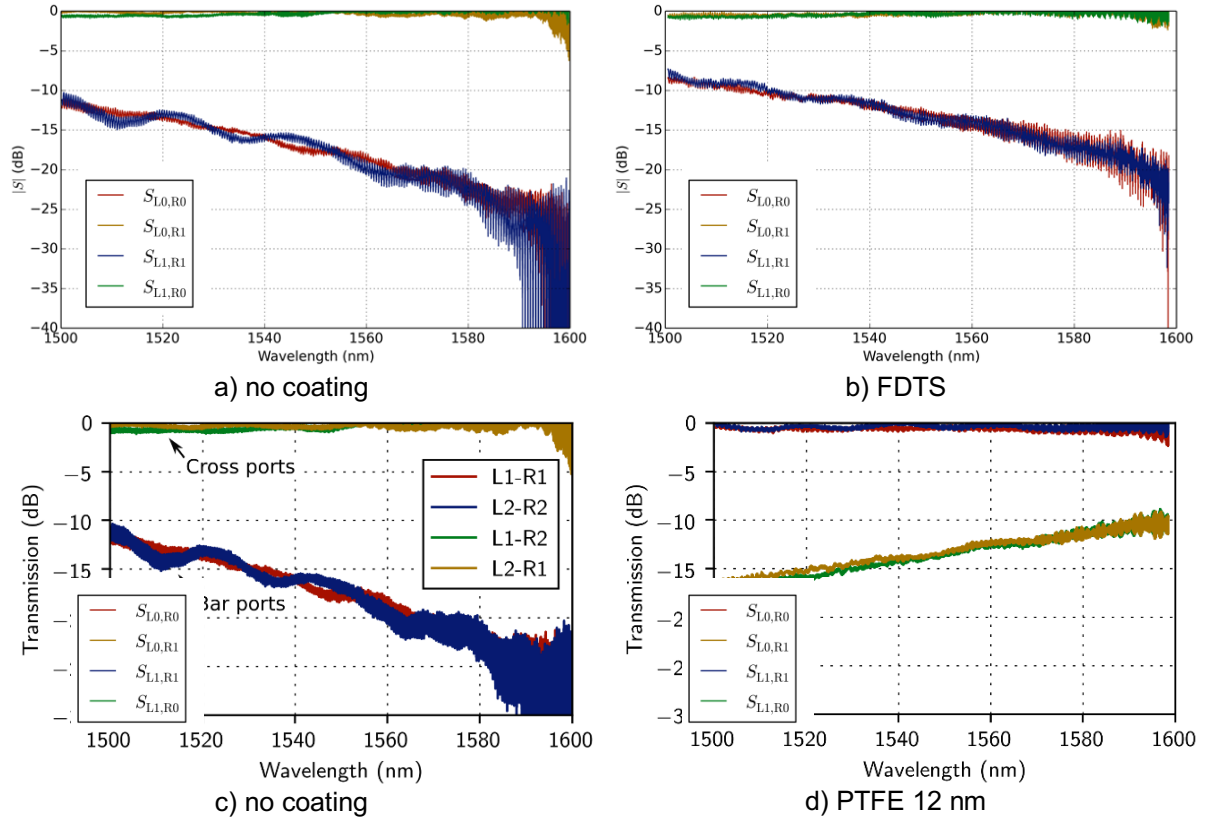


Fig. 5: Transmission measurements of FDTD and PTFE coated optical couplers compared to the uncoated coupler, respectively

## 4. Conclusions and outlook

The FDTD monolayer was found to be an applicable coating for the EWOD controlled active optical switch. This paper presented its suitability regarding the optical compatibility, EWOD actuation and long-term stability. As anti-sticking layer applied to the active optical surface, it enables the EWOD driven liquid motion over the coupler and transmission measurements validate that the effect on the optical performance is insignificant. Long-term stability of the coating in contact with the liquids HPC and TPS has been proved experimentally. The thickness of the layer corresponds to the length of the molecule which is about 1 nm. Contact angle measurements show that the dewetting properties of the FDTD are comparable to PTFE. However, the FDTD vapor phase deposition exhibits significant advantages as it leads to a substantial thinner film that forms stable chemical bonds to the Si/SiO<sub>2</sub> surfaces of the optical coupler whereas the thinnest possible dip coated PTFE layer has a thickness of 12 nm. Furthermore, the substrate materials for FDTD application are not limited to Si or SiO<sub>2</sub> as the silane can bond covalently to any hydroxylated surface.

The positive results indicate that the FDTD might also be a suitable coating for the active EWOD structures. The fluidic substrates comprise channels with a height of 100 μm. Thus, the uniform coating by FDTD vapor deposition would also be advantageous for the EWOD component of the switch. Sample preparation and experimental verification of FDTD coated fluidic substrates are ongoing.

## Acknowledgements

This work has been supported by the SwIFT project, funded by the EC 7th Framework Program with the project reference of 619643 and was carried out in collaboration with CommScope Inc. Furthermore the authors would like to thank Cristina Lerma Arce for the supportive work.

## References

- [1]. Self-assembled monolayers as anti-stiction coatings for MEMS- characteristics and recent developments(2000)
- [2]. Beck - Improving stamps for 10 nm level wafer scale nanoimprint(2002)
- [3]. Schiff - Controlled co-evaporation of silanes for nanoimprint stamps(2005)
- [4]. Anti-adhesive effects of diverse self-assembled monolayers in nanoimprint lithography(2007)
- [5]. Si - The NanoTuFe — Fabrication of large area periodic nanopatterns with tunable feature sizes at low cost(2017)
- [6]. Gu, H., Duits, M. H. G., and Mugele, F., "A hybrid microfluidic chip with electrowetting functionality using ultraviolet (UV)-curable polymer," *Lab on a chip*, vol. 10, no. 12, pp. 1550–1556, 2010
- [7]. Adzima, Brian J.; Velankar, Sachin S. (2006): Pressure drops for droplet flows in microfluidic channels. In *J. Micromech. Microeng.* 16 (8), pp. 1504–1510. DOI: 10.1088/0960-1317/16/8/010.
- [8]. Handique, K.; Burke, D. T.; Mastrangelo, C. H.; Burns, M. A. (2000): Nanoliter Liquid Metering in Microchannels Using Hydrophobic Patterns. In *Anal. Chem.* 72 (17), pp. 4100–4109. DOI: 10.1021/ac000064s.
- [9]. Herbert D'heer, Cristina Lerma Arce, Stijn Vandewiele, Jan Watté, Koen Huybrechts, Roel Baets, and Dries Van Thourhout, "Nonvolatile Liquid Controlled Adiabatic Silicon Photonics Switch," *J. Lightwave Technol.* 35, 2948-2954 (2017)
- [10]. H. D'heer, C. L. Arce, J. Watté, K. Huybrechts, R. G. F. Baets, and D. Van Thourhout, "Broadband and Non-volatile Liquid Controlled Silicon Photonics Switch," in *Conference on Lasers and Electro-Optics*, OSA Technical Digest (2016) (Optical Society of America, 2016), paper SM3G.6.
- [11]. S. Günther et al., "EWOD system designed for optical switching," 2017 IEEE 30th International Conference on Micro Electro Mechanical Systems (MEMS), Las Vegas, NV, 2017, pp. 1329-1332. doi: 10.1109/MEMSYS.2017.7863665
- [12]. Yuan, Yuehua; Lee, T. Randall (2013): Contact Angle and Wetting Properties. In: Gianangelo Bracco und Bodil Holst (Hg.): *Surface Science Techniques*, Bd. 51. Berlin, Heidelberg: Springer Berlin Heidelberg, S. 3–34
- [13]. Moon, Hyejin; Cho, Sung Kwon; Garrell, Robin L.; Kim, Chang-Jin "CJ" (2002): Low voltage electrowetting-on-dielectric. In: *Journal of Applied Physics* 92 (7), S. 4080–4087. DOI: 10.1063/1.1504171.
- [14]. Barbulovic-Nad, Irena; Yang, Hao; Park, Philip S.; Wheeler, Aaron R. (2008): Digital microfluidics for cell-based assays. In: *Lab on a chip* 8 (4), S. 519–526. DOI: 10.1039/b717759c.

- [15]. Koo, Bonhye; Kim, Chang-Jin (2013): Evaluation of repeated electrowetting on three different fluoropolymer top coatings. In: J. Micromech. Microeng. 23 (6), S. 67002. DOI: 10.1088/0960-1317/23/6/067002.
- [16]. Terrab, Soraya; Watson, Alexander M.; Roath, Christopher; Gopinath, Juliet T.; Bright, Victor M. (2015): Adaptive electrowetting lens-prism element. In: Optics express 23 (20), S. 25838–25845. DOI: 10.1364/OE.23.025838.
- [17]. Sharangpani, R.; Singh, R.; Drews, M.; Ivey, K. (1997): Chemical vapor deposition and characterization of amorphous teflon fluoropolymer thin films. In: Journal of Electronic Materials 26 (4), S. 402–409. DOI: 10.1007/s11664-997-0110-z.
- [18]. Biederman, H. (2000): RF sputtering of polymers and its potential application. In: *Vacuum* 59 (2-3), S. 594–599. DOI: 10.1016/S0042-207X(00)00321-3.